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NEUTRON IRRADIATION EFFECTS ON FATIGUE CRACK PROPAGATION IN TYP--ETC(U)

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crack propagation mode of the specimens which exhibited increased crack propagation rates was primarily intergranular while a transgranular mode was observed for specimens with lower crack propagation rates. The results point toward a synergistic relationship between thermomechanical history, precipitate formation, and hold time effects as the responsible mechanism for the crack propagation performance.



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NEUTRON IRRADIATION EFFECTS ON FATIGUE CRACK PROPAGATION IN TYPE 316 STAINLESS STEELS AT 649°C

INTRODUCTION

The consideration of the effects of fast neutron irradiation on the elevated temperature properties and performance of reactor structural materials has been recognized as an important design criteria by both reactor designers and safety analysts. Since it is known that these materials will be subjected to cyclic and combined cyclic-static loads during irradiation, the effects of the irradiation environment on the crack propagation performance of the materials must be known.

The effects of fast neutron irradiation on the fatigue crack propagation performance of the austenitic stainless steels have been discussed in previous reports [1-4]. The results show that, for fluences up to approximately 2×10^{22} n/cm² (>0.1 MeV) and temperatures up to 649°C, neutron irradiation had no significant influence on fatigue crack propagation rate during continuous cycling of annealed stainless steel when compared with unirradiated test results. The inclusion of a one minute tensile hold period in the fatigue cycle was found to significantly increase the crack propagation rates in the annealed steel. For steel which had been cold worked prior to irradiation, increased crack propagation rates were observed during both continuous and hold time fatigue cycles.

The purpose of this report is to review new results from a study of the effects of fast neutron irradiation to fluence levels of approximately 5×10^{22} n/cm² (0.1 MeV) at 649°C on the crack propagation performance of Type 316 stainless steel during continuous and hold time fatigue cycling. The test results are compared with the lower fluence data at 649°C [4] and with results at 593°C. Scanning electron microscopy was used to investigate the failure mode in the irradiated and unirradiated specimens as an aid to the interpretation of the crack propagation results.

EXPERIMENTAL PROCEDURES

The fatigue crack propagation specimen dimensions and the chemical composition of the Type 316 stainless steel plate material have been previously reported [3,5]. Single-edge-notched (SEN) cantilever specimens were prepared from the plate material such that the plane of crack propagation was perpendicular to the plate rolling direction. For neutron irradiation purposes, SEN center sections, 3.81 cm by 6.36 cm, were irradiated in an NRL controlled temperature subassembly (X-200) at 649°C in the Experimental Breeder Reactor No. II (EBR-II) to calculated fast neutron fluences from 4.5 to 5.1×10^{22} n/cm² (>0.1 MeV). After irradiation, end tabs were welded in-cell to the center sections to produce the final specimen configuration. Previous experimental work has shown that crack propagation behavior in similarly welded specimens was the same as that in non-welded specimens [6].

Complete details of the test and data analysis procedures have been reported [1,3,7]. Briefly, all tests were conducted using a zero-to-tension loading cycle at 0.17 Hz (10 cpm) to a constant maximum load in air. Hold time effects were investigated for certain tests by maintaining the maximum tensile load constant for selected time periods during each cycle. Induction heating was employed to achieve a test temperature of 649°C with control maintained to within +3°C. Crack lengths were measured periodically using a remote, high-resolution television system for the irradiated tests. The experimental data were analyzed to yield crack propagation rate, da/dN , as a function of the stress intensity factor range, ΔK , according to the relationship

$$da/dN = C(\Delta K)^m \quad (1)$$

where C and m are constants dependent on test parameters, the material and environmental factors. It is well established that this relationship describes the fatigue crack propagation behavior of metallic materials under a wide variety of experimental conditions.

Scanning electron microscopy (SEM) was used to investigate the crack propagation mode of all specimens tested at 649°C. Specimen sections containing one fracture surface were prepared from both the unirradiated and the irradiated fatigue specimens. The unirradiated specimen sections were examined using a Coates and Welter Cwikscan microscope. The irradiated specimen sections were examined using an I.S.I. Super II microscope modified for remote hot cell operation. All fracture surfaces were examined in the high voltage operating range at the centerline of the fracture (midthickness of the specimen) starting at the root of the machined notch.

EXPERIMENTAL RESULTS

The effects of neutron irradiation and hold time on the fatigue crack propagation behavior of annealed and cold worked Type 316 stainless steel at 649°C are presented in Figs. 1 through 4 together with previous results obtained at 593°C [3,5] and at 649°C [4]. The results of fractographic examination of the tested specimens are presented in Figs. 5 through 8. All experimental results are summarized in Table 1.

For the continuous cycling (zero hold time) test conditions, Fig. 1 shows that irradiation at 649°C to 4.9×10^{22} n/cm² produced no significant effect on the crack propagation rate in annealed steel when compared with results at 593°C. However, in the cold worked material, fatigue crack propagation behavior was highly sensitive to neutron irradiation and fluence. The cold worked material irradiated at 649°C to a fluence of 1.2×10^{22} n/cm² exhibited a greater increase in crack propagation rate than previously shown at 593°C. Interestingly, when irradiated at a higher fluence of 5.1×10^{22} n/cm², crack propagation rates were decreased nearly to the level of the similar unirradiated values at 593 and 649°C.

The results for the effect of a one minute hold time in the annealed and cold worked material at 649°C are shown in Figs. 3 and 4 and suggest a marked dependence of crack propagation rate on irradiation exposure, hold time and test temperature for both material conditions. In Fig. 3, the crack propagation rate of the annealed steel irradiated to 1.2×10^{22} n/cm² and tested at 649°C with a one minute hold time was greatly increased when compared with the results of 593°C. The inclusion of a two minute hold time on the fatigue cycle for the steel irradiated to 4.9×10^{22} n/cm² produced a further increase in the crack propagation rate at all levels of ΔK for tests at 649°C. Furthermore, it is important to note that the slope of both the unirradiated and

Table 1 - Summary of Experimental Results at 649°C

Material Condition	Neutron Fluence ($\times 0.1$ MeV)	Hold Time at Maximum Cyclic Load (min.)	Effect on Crack Propagation Rate†	Effect on Crack Propagation Mode
Annealed	0	0	no effect	transgranular
	1.2, 1.4	0	no effect	mixed*
	4.9	0	no effect	mixed
	0	1	no effect	transgranular + mixed**
	1.2	1	large increase	intergranular
	4.8	2	large increase	intergranular
20% Cold Worked	0	0	no effect	transgranular
	1.2	0	small increase	intergranular
	5.1	0	large decrease	mixed
	0	1	large decrease	transgranular
	1.4	1	large increase	intergranular
	4.5	1	small increase	intergranular

† Relative to results from unirradiated and irradiated tests respectively, at 593°C (See Figs. 1 through 4).

* Mixed = transgranular + intergranular

** Transgranular: $\Delta K < 30 \text{ MPa}\sqrt{\text{m}}$.

Mixed: $\Delta K \geq 30 \text{ MPa}\sqrt{\text{m}}$.

irradiated da/dN vs ΔK results at 649°C are significantly higher than for the results at 593°C . This change in slope suggests that the failure mechanism in the annealed material at 649°C during hold time tests may have been different than at 593°C .

For the cold worked steel, Fig. 4, the inclusion of a one minute hold time substantially increased the crack propagation rate at 649°C after irradiation to 1.4×10^{22} n/cm^2 , while further irradiation to 4.5×10^{22} n/cm^2 produced a crack propagation rate which was nearly the same as the irradiated results at 593°C . The significant effect of hold time on the cold worked steel can be seen by comparison of Figs. 2 and 4. The observation of a decrease in crack propagation rate with increased fluence for the hold time results at 649°C is consistent with the behavior seen for the cold worked material under purely cyclic conditions, Fig. 2. In particular, the results observed in the irradiated steel at 649°C suggest that the effects of hold time and irradiation on crack propagation rate in the cold worked steel saturate at this temperature for fluences between 1.4 and 4.5×10^{22} n/cm^2 . This point is of considerable interest from the reactor design viewpoint since component lifetimes could be severely limited where hold times are expected to be encountered at low fluence levels.

Previous fractographic examinations of failure mode in unirradiated, annealed and cold worked Type 316 stainless steel, fatigue tested at 593°C , revealed that cracks propagated transgranularly for zero hold time tests and intergranularly for one minute hold time tests [5]. The results for the annealed, unirradiated steel tested at 649°C are shown in Figs. 5a and 5b for comparison with the scanning electron microscope examinations of fracture surfaces formed during fatigue crack propagation in the cold worked, unirradiated steel tested at 649°C , Figs. 5c and 5d. It can be seen that for zero hold time test conditions the crack propagation mode in the annealed material was primarily transgranular at 649°C , Fig. 5a. However, an intergranular failure mode was not observed at ΔK 's below $30 \text{ MPa}\sqrt{\text{m}}$ for one minute hold time tests at 649°C , Fig. 5b. At ΔK levels above this value, the crack propagated by a mixture of transgranular and intergranular fracture. In the cold worked condition, the failure mode at 649°C also was primarily transgranular or mixed for zero and one minute hold time tests, respectively, Figs. 5c and 5d.

The crack propagation failure modes of the low and high fluence irradiated fatigue crack propagation specimens tested at 649°C are illustrated in Figs. 6 and 7. For the annealed, irradiated steel, Figs. 6a and 6c show that the crack propagation in the continuous cycling (zero hold time) specimens was by a mixture of both transgranular and intergranular modes at 649°C . An entirely intergranular mode of crack propagation was observed in the annealed, irradiated specimens tested with hold times of one and two minutes, Fig. 6b and 6d, respectively. The SEM micrographs for the cold worked, irradiated steel show that, at the lower fluence level, the crack propagation mode was predominantly intergranular regardless of hold times, Figs. 7a and 7b. At the higher fluence level, the crack propagation occurred by a mixture of both transgranular and intergranular modes, Fig. 7c, for the continuous cycling specimen and became entirely intergranular for the specimen tested with a one minute hold time, Fig. 7d.

A detailed examination was conducted of the irradiated crack propagation specimens tested with hold times to determine whether there was any evidence of intergranular cavity formation and/or linkage. For the one minute hold time specimens (Figs. 6b, 7b, and 7d), no evidence of intergranular cavities was found. However, the specimen tested with a two minute hold time (Fig. 6d) was found to have intergranular cavities associated with precipitate particles as shown in Fig. 8. The cavity size is seen to be of the order of $1 \mu\text{m}$ and with cavity spacing from 3 to $8 \mu\text{m}$. The observation of further details of the intergranular cavities was effectively obscured by the adherent oxide layer

on the fracture surfaces. However, since the specimens were irradiated and tested at 649°C, it is possible that the relatively small amount of helium produced in the specimens (≤ 10 ppm) may have been influential in promoting cavity nucleation at hold times shorter than those which have been observed in unirradiated specimens [8].

DISCUSSION

The results from fatigue tests of the annealed steel show that irradiation to both fluence levels at 649°C produced no significant effect on the crack propagation rate when compared with unirradiated steel tested at 649°C. However, for the 20 percent cold worked steel, irradiation to the lower fluence level increased the crack propagation rate while at the higher fluence the crack propagation rate was only slightly increased relative to the unirradiated material. For tests conducted using creep-fatigue cycling, the effect of a one minute hold time at the maximum cyclic load was to produce a marked increase in the crack propagation rate of the annealed steel irradiated to the lower fluence level. At the higher fluence level, the inclusion of a two minute hold time in the creep-fatigue cycle resulted in a further increase in the crack propagation rate. The effect of a one minute hold time in the irradiated, 20 percent cold worked steel was to significantly increase the crack propagation rates, particularly at the low fluence level at 649°C. These results will be discussed with respect to the SEM observations to develop further insight into the effects of irradiation on fatigue crack propagation.

Previous thermal aging studies [5,8] of unirradiated Type 316 stainless steel have shown that aging at 593°C reduced the effects of hold time and delayed the transition from a transgranular to an intergranular failure mode to the hold times longer than one minute. Other studies [4] have shown that considerable precipitation occurs during testing at 649°C for the steel used in the present work, but not 593°C during testing. The similar behavior seen in the present study for the unaged and unirradiated, annealed and cold worked Type 316 stainless steel tested at 649°C with a one minute hold time suggests that the carbide precipitation which occurred during testing was responsible for the reduction in crack propagation rates to levels equivalent to those in aged material at 593°C. Furthermore, these results indicate that, despite the higher test temperature, the more rapid carbide precipitation kinetics reported for Type 316 stainless steel at temperatures above 600°C [9,10] were responsible for maintaining a transgranular or mixed failure mode throughout the 649°C one minute hold time tests in the unirradiated steel despite the higher slope of the da/dN vs ΔK curves.

Comparison of Figs. 5a, 5b and 6 clearly shows that the transgranular failure mode observed in the annealed, unirradiated steel at 649°C was not seen in the irradiated steel. This provides strong evidence that the intergranular precipitate mechanism operative in the unirradiated steel was influenced by the irradiation. Previous studies of 316 stainless steel irradiated to both low and high fluences at temperatures from 400 to 650°C have shown that the influence of irradiation on phase instability is quite pronounced, resulting in a variety of new precipitate phases [11-13] which include modified forms of the $M_{23}C_6$ precipitate. Further, comparison of the results in Figs. 1 and 3 shows that the effect of irradiation on the crack propagation performance of the annealed steel was significant only when a hold time was included in the fatigue cycle. In particular, it is seen that the slope of the two minute hold time, da/dN vs ΔK curve is identical to that for the one minute hold time result at 649°C despite the shift to lower values of K at equivalent values of da/dN . This suggests that the increased crack propagation rate for the hold time tests of the annealed, irradiated steel was the result of a failure mechanism which was different than that for the continuous cycling tests. This is supported by the SEM results which show that intergranular cavity formation occurred on the failure surface of the two minute hold time specimen, Fig. 8, but was

not observed for the continuous cycling specimens. The very similar intergranular failure mode appearance of the two and one minute hold time specimens, Figs. 6b and 6d, suggests that a tendency toward cavity formation may have been prevalent in the one minute hold time specimen despite the absence of direct fractographic evidence for the presence of cavities.

For the cold worked specimens irradiated to the lower fluence level, the predominantly intergranular mode of crack propagation evident in Figs. 7a and 7b is in agreement with the higher crack propagation rates for the irradiated, zero and one minute hold time specimens in Figs. 2 and 4. This suggests that the intergranular precipitation which was shown to be effective in reducing the crack propagation rate and maintaining a transgranular failure mode in the unirradiated, cold worked steel was influenced by the irradiation. However, for the cold worked specimen irradiated to the higher fluence level and tested with zero hold time, the markedly lower crack propagation rate in Fig. 2 and the mixed transgranular-intergranular failure mode in Fig. 7c strongly suggest that the higher fluence may have promoted intergranular precipitate formation to suppress the crack propagation rate and the transition to an intergranular cracking mode. For the cold worked specimen irradiated to the higher fluence level and tested with a one minute hold time, the reduction of the crack propagation rate in Fig. 2 to the level observed for the low fluence 593°C specimen also suggests that intergranular precipitate formation, possibly induced by the irradiation, was responsible for the lowered crack propagation rate at 649°C. This is supported by the tendency of the failure mode in Fig. 7d toward a more transgranular appearance when compared with the intergranular failure mode in Fig. 7b.

The fractographic features and the crack propagation results, for both the annealed and cold worked 316 steel irradiated to two fluence levels at 649°C and tested at the same temperature, point toward a synergistic relationship between thermomechanical history, precipitate formation and hold time effects as the mechanism responsible for the crack propagation performance under the conditions studied. The evidence shows that the pre-irradiation thermomechanical history is critical since it provided the basic microstructure which was influenced by the elevated temperature irradiation in terms of the suggested fluence dependent precipitate formation. The irradiation microstructure, in turn, influenced the crack propagation rate and mode, by means of thermally activated processes, depending on the fatigue cycle applied (continuous cycling or hold time). The results show that the cold worked steel exhibits the largest susceptibility to the effects of irradiation, temperature and hold time while the effects of irradiation on the fatigue properties of the annealed steel were seen only when hold time was included in the fatigue cycle. Nevertheless, additional studies of precipitate formation in these test specimens, now underway, will be necessary to confirm the suggested role of the precipitates on the crack propagation performance.

SUMMARY AND CONCLUSIONS

The effects of neutron irradiation at 649°C to two fluence levels on the fatigue crack propagation performance of annealed and 20 percent cold worked Type 316 stainless steel were investigated for continuous cycling and hold time conditions at 649°C. The following conclusions are drawn from the results:

1. Neutron irradiation at 649°C has no significant effect on fatigue crack propagation rate in annealed Type 316 stainless steel during continuous cyclic loading when compared with unirradiated tests at this temperature.

2. The inclusion of tensile hold periods significantly increased the crack propagation rates in annealed Type 316 stainless steel neutron irradiated and tested at 649°C when compared to both unirradiated results at 649 and 593°C and neutron irradiated results at 593°C.

3. For 20 percent cold worked Type 316 stainless steel, neutron irradiation at 649°C to fluences of approximately 1 to 2×10^{22} n/cm² increased crack propagation rate during both continuous cycling and hold time tests when compared to both unirradiated results at 649 and 593°C and neutron irradiated results at 593°C. However, neutron irradiation to approximately 5×10^{22} n/cm² at 649°C produced a smaller increase in the crack propagation rate of 20 percent cold worked steel during both continuous cycling and hold time tests.

4. In the unirradiated, annealed and cold worked one minute hold time tests at 649°C, the reduced crack propagation rate, relative to that at 593°C, was accompanied by a purely transgranular failure mode at ΔK levels less than 30 MPa√m and a significantly higher da/dN vs ΔK slope. It is concluded that the carbide precipitation which occurred during testing acted to delay the transition to a mixed intergranular-transgranular failure mode at a ΔK of 30 MPa√m and to reduce the crack propagation rate in the unirradiated steel.

5. Based on the fractographic observations of the irradiated specimens, it is concluded that thermally activated processes, such as those concerned with intergranular precipitate formation and with cavity formation and growth, were responsible for the increased crack propagation rates in both the annealed and cold worked specimens at 649°C when hold periods were included in the fatigue cycle. The fractographic results also suggest that intergranular precipitate formation, in the absence of the development of cavities, may have acted to suppress the crack propagation rates for the cold worked steel during continuous cycling and hold time tests following irradiation to the approximately 5×10^{22} n/cm² fluence level.

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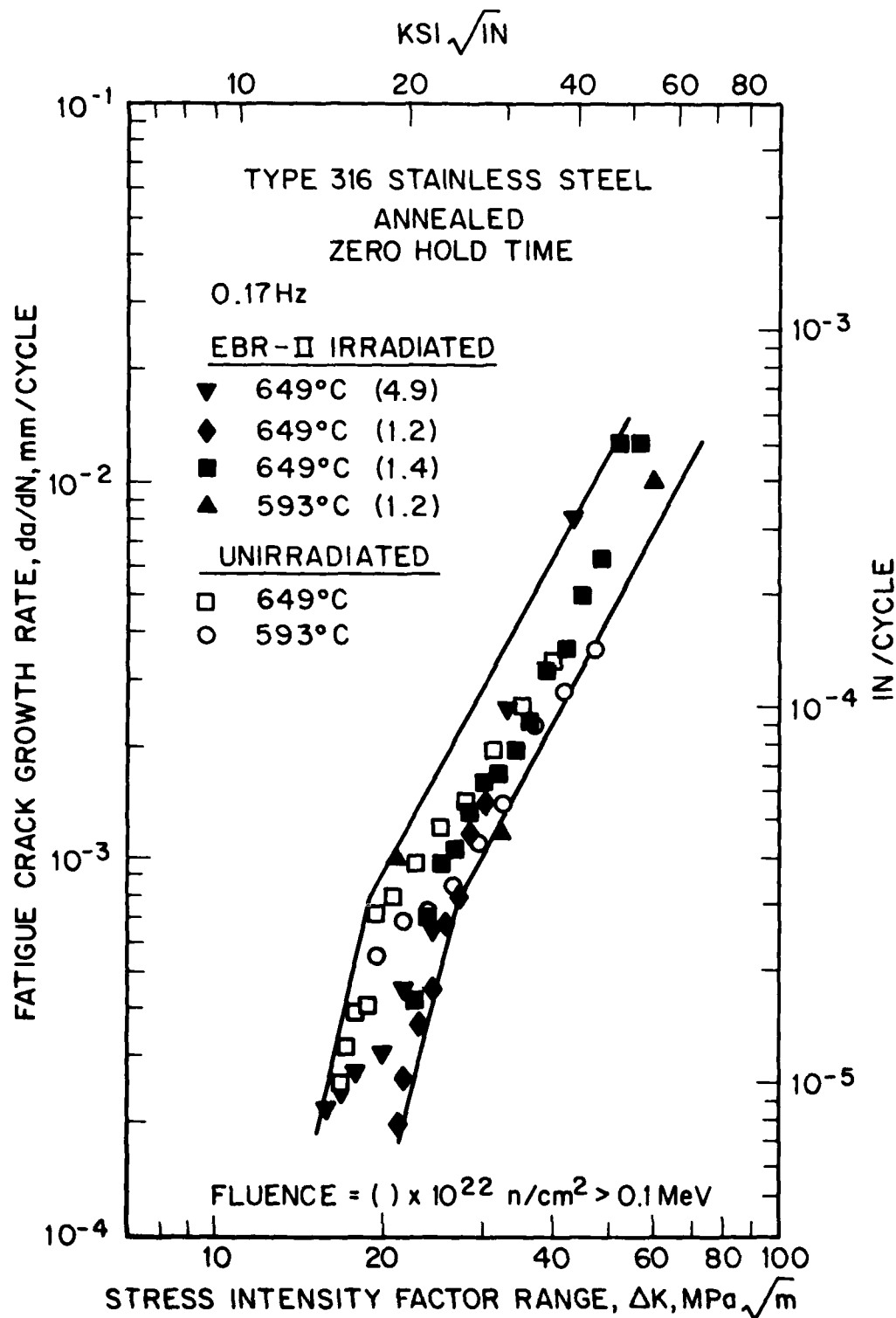


Fig. 1 — Comparison of fatigue crack propagation rates, da/dN , in air at 593 and 649°C for annealed, EBR-II irradiated and unirradiated Type 316 stainless steel tested with continuous cyclic loading at 0.17 Hz (10 cpm).

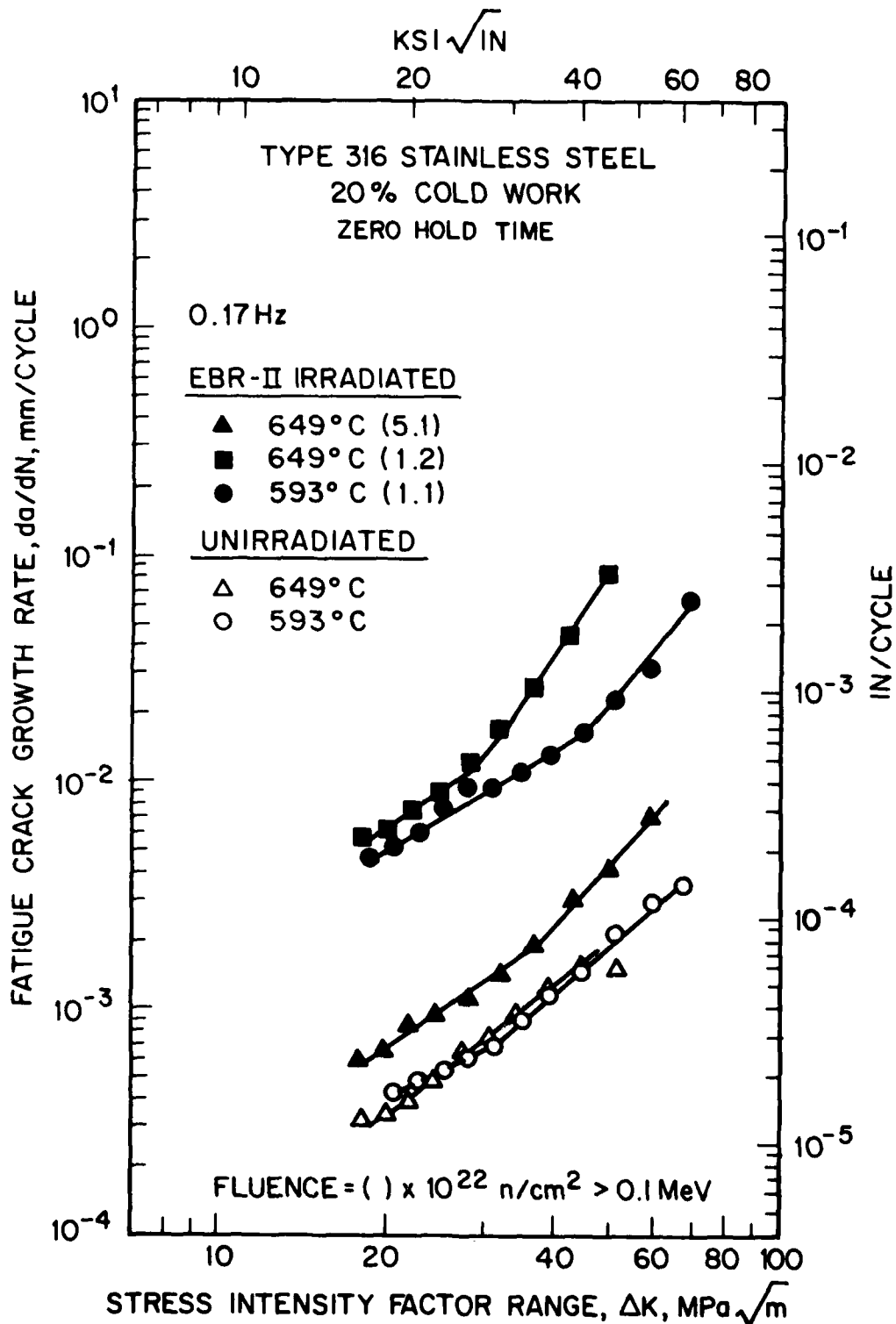


Fig. 2 — Comparison of fatigue crack propagation rates, da/dN in air at 593 and 649°C for 20 percent cold worked, EBR-II irradiated and unirradiated Type 316 stainless steel tested with continuous cyclic loading at 0.17 Hz (10cpm).

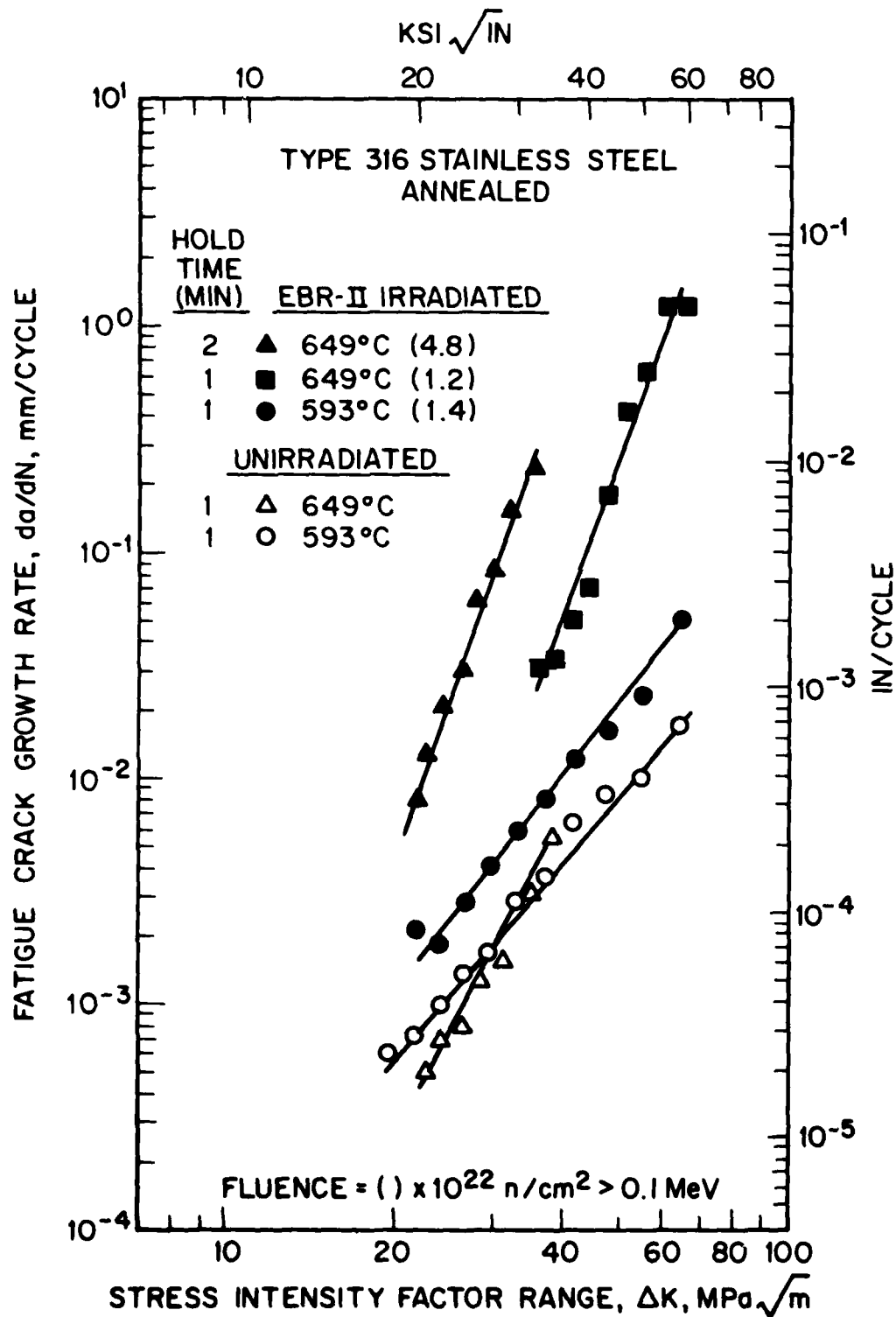


Fig. 3 - Comparison of fatigue crack propagation rates, da/dN , in air at 593 and 649°C for annealed, EBR-II irradiated and unirradiated Type 316 stainless steel tested with hold times.

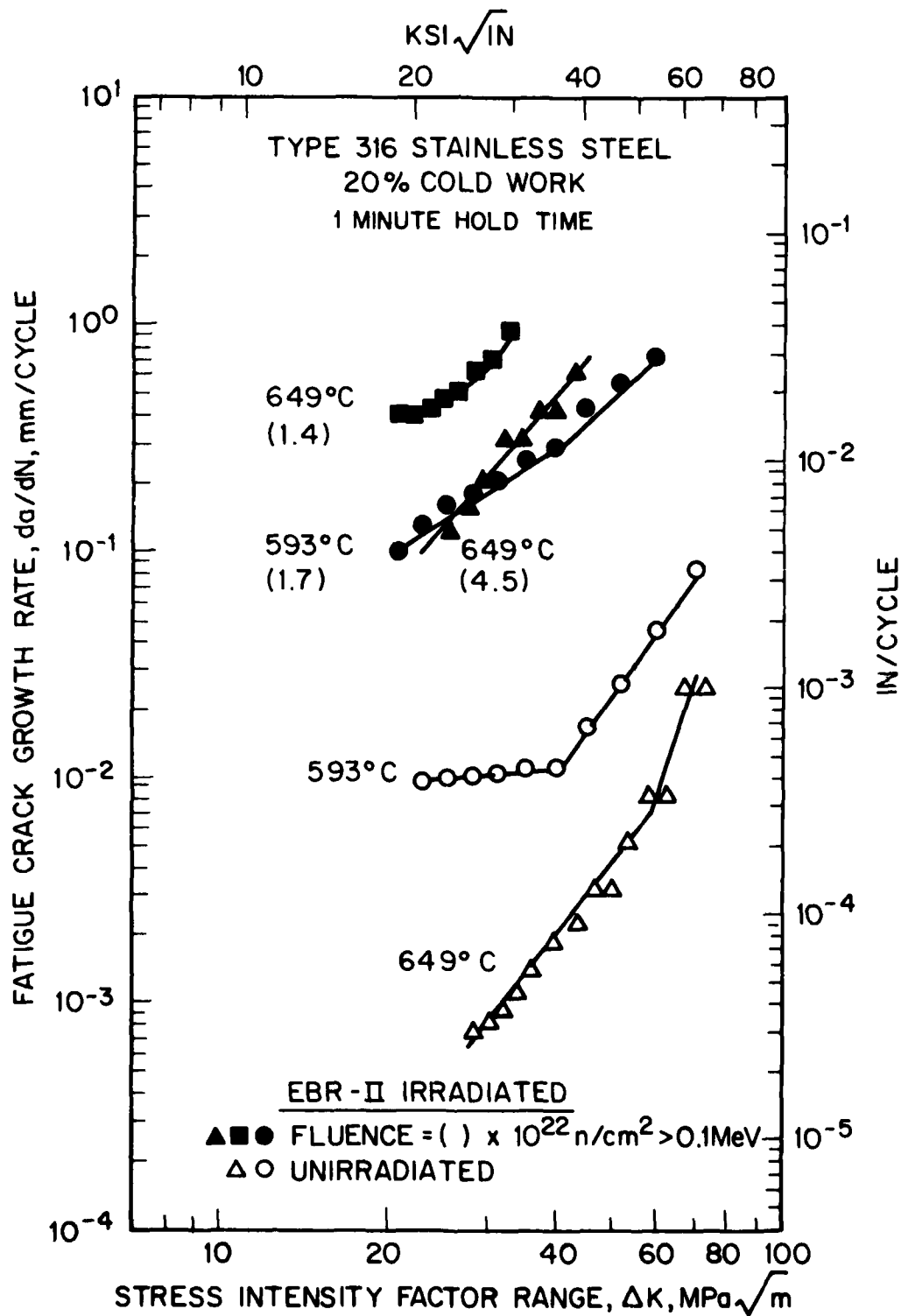


Fig. 4 — Comparison of fatigue crack propagation rates, da/dN , in air at 593 and 649°C for 20 percent cold worked, EBR-II irradiated and unirradiated Type 316 stainless steel tested with hold times.

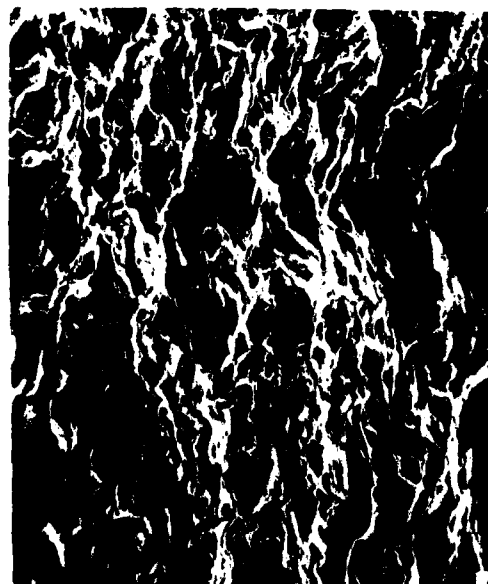
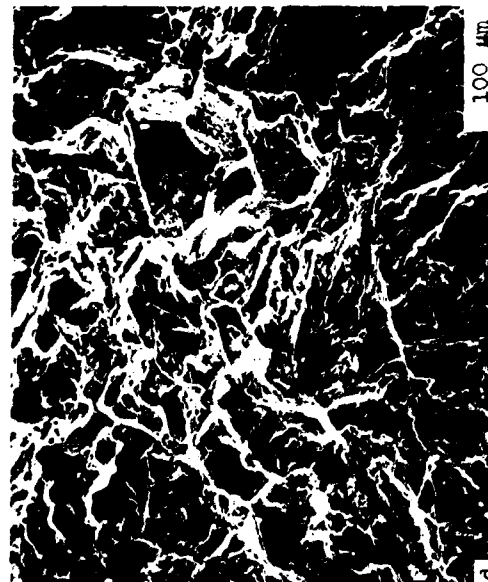


Fig. 5 — Scanning electron micrographs of the crack propagation mode of annealed and 20-percent cold worked, unirradiated Type 316 stainless steel at 649°C: (a) annealed, zero hold time; (b) annealed, one minute hold time; (c) 20-percent cold worked, zero hold time; (d) 20-percent cold worked, one minute hold time.

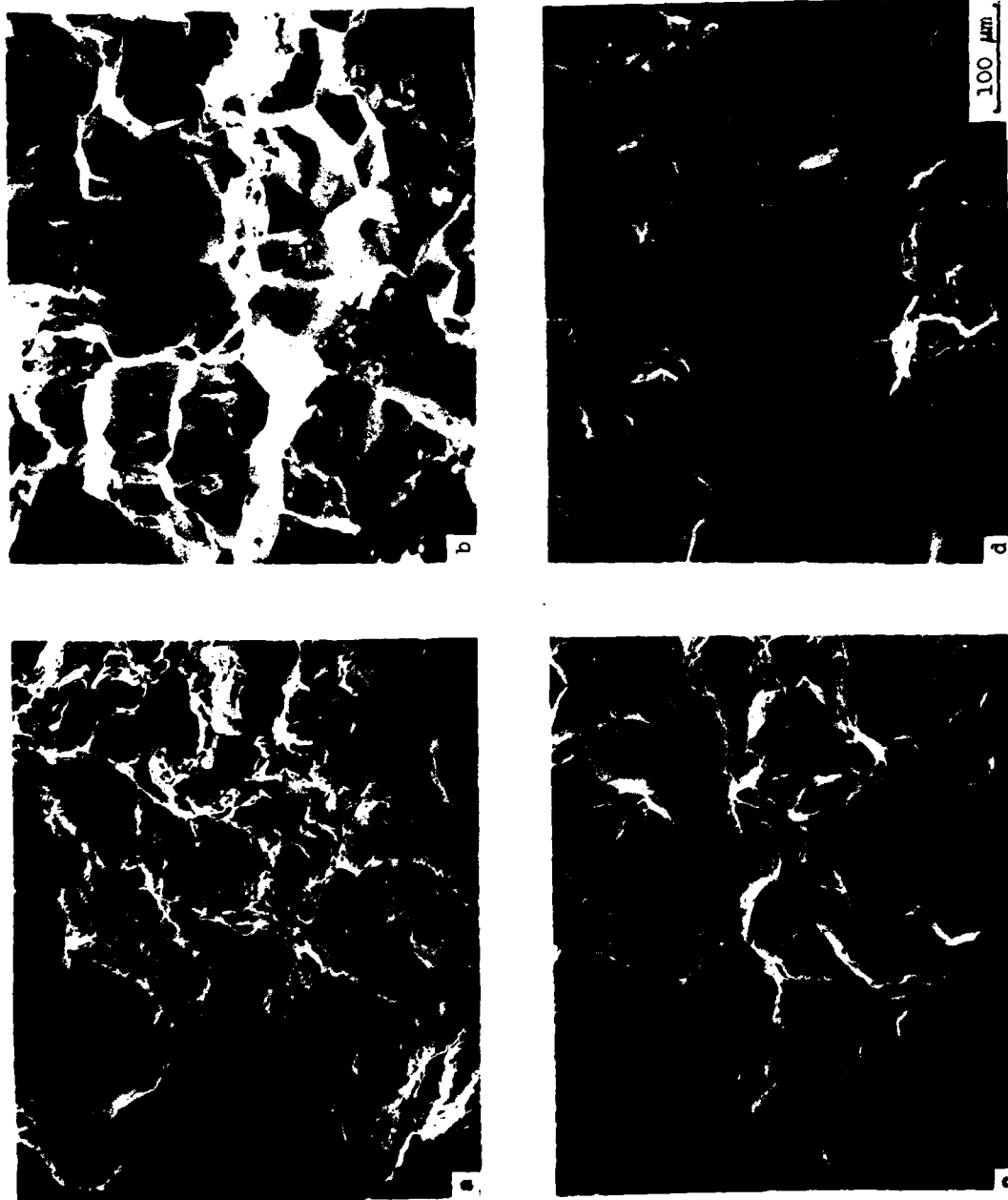
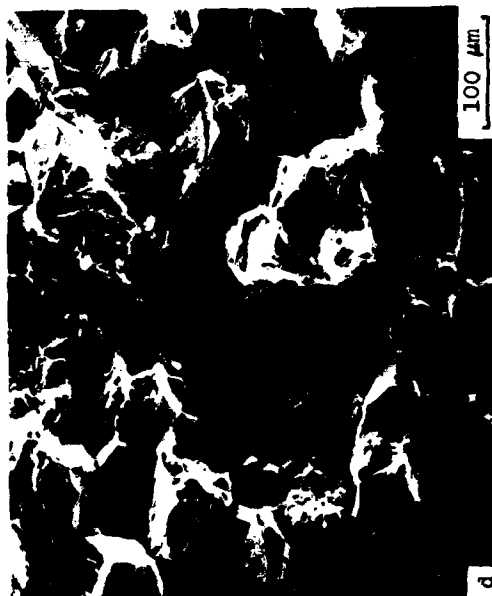


Fig. 6 — Scanning electron micrographs of the crack propagation mode of annealed Type 316 stainless steel irradiated and tested at 649°C: (a) zero hold time, 1.2×10^{22} n/cm²; (b) one minute hold time, 4.9×10^{22} n/cm²; (c) zero hold time, 4.8×10^{22} n/cm²; (d) two minute hold time, 4.8×10^{22} n/cm².



a



b



c



d

Fig. 7 — Scanning electron micrographs of the crack propagation mode of 20-percent cold worked stainless steel irradiated and tested at 649°C: (a) zero hold time $1.2 \times 10^{22} \text{ n/cm}^2$; (b) one minute hold time $1.4 \times 10^{22} \text{ n/cm}^2$; (c) zero hold time, $5.1 \times 10^{22} \text{ n/cm}^2$; (d) one minute hold time, $4.5 \times 10^{22} \text{ n/cm}^2$.



a



b

20 μm

Fig. 8 — Scanning electron micrographs of intergranular cavities associated with precipitates in annealed Type 316 stainless steel irradiated to 4.8×10^{22} n/cm² at 649°C and tested with a two-minute hold time at 649°C.